IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Sisson, et. al.) Docket No. MGP.P.US0081) Art Unit: 1714
For: Article Comprising Light Absorbent Composition to Mask Visual Haze and Related Methods	Examiner: Bruenjes, C.)
Serial No. 10/769,167)
Filed: 1/30/2004););)
Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450	
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DECLARATION #2 OF DR. KEVIN L. ROLLICK

- I, Kevin L. Rollick, Ph.D., hereby declare that:
- 1. I am a resident of Monroe Falls, Ohio and am employed as a Senior Research Scientist at M&G Polymers, USA. I am not an inventor of the subject matter disclosed or claimed in the subject patent application.
- 2. My educational background includes a Bachelor of Science from Indiana University of Pennsylvania (cum laude, 1975); and a Ph.D. in Organic Chemistry and minor in Inorganic Chemistry from Indiana University at Bloomington, Indiana (cum laude, 1981).

- 3. I am a named inventor in 13 United States letters patents, six of which relate to the field of polyester polymers.
- 4. I have been employed by M&G Polymers USA, LLC, and its predecessors (The Goodyear Tire & Rubber Company and Shell Oil Company) for approximately twenty six (26) years and have worked in the field of polymer resins for sixteen (16) years. I am presently a Senior Research Chemist at M & G Polymers USA, LLC's research facility located in Sharon Center, Ohio.
- 5. I have authored or co-authored seven technical papers, one of which was awarded the Best Paper Award.
- 6. For the last 8 years I have been a member of research team charged with developing barrier bottles for beer and juice applications. During this development, I conducted and observed many polyester/MXD6 blends, both with and without cobalt, and with and without colorants. These blends have been injected into preforms and blown into reheat stretch blown bottles.
- 7. I have reviewed the Office Action stating that the colors of the MXD6/PET blends are from the nylon domains reflecting light.
- 8. I have read Kim '987 and conclude that it does not teach that the color of the composition is caused by nylon domains. Rather, I conclude that Kim '987 teaches that the color is caused by the presence of catalyst residues in the PET. I conclude this because Kim '987 teaches that the inherent greenish color can be neutralized by changing the PET to a PET produced from a catalyst system based on germanium and antimony or having no residual manganese or cobalt as opposed to PET produced from a catalyst system based on antimony,

cobalt and manganese (col 3, Lines 45 – 54).

- 9. I conclude that the data of Kim '987 also teach that the catalyst as the variable affecting color at col 4, lines 4-21. In addition, the Examples demonstrate that different PET's have a different color without the addition of cobalt octoate. This means that the catalyst is the cause of the color, not the domains.
- 10. The following table breaks down the examples showing the impact of the catalyst. I have recast the data from Kim '987 (cols 7, lines 20 55) in the following table. As can be seen the change of catalyst (A and B) show a color shift. C1 and C2 are green even with cobalt, D1 is yellowish as opposed to green because of the shift in the PET catalyst. Only D3 is considered neutral. The rest of the compositions have some type of color.

Table I

Blend	PET CATA	PET CATALYST COMPOSITION				Co	Color
					(wt %)	Octoate	
						Ppm	
	Antimony	Cobalt	Manganese	Germanium			
A	95	62	62	***************************************	7.5		Green
В	4	<0.1	<0.1	8	7.5	-	Yellowish
C1	95	62	62	,	7.5	100	Green
C2	95	62	62		7.5	150	Green
D1	4	<0.1	<0.1	8	7.5	50	Yellowish
D2	4	<0.1	<0.1	8	7.5	100	Less
					-		Yellowish
D3	4	<0.1	<0.1	8	7.5	150	Neutral
D4	4	<0.1	<0.1	8	7.5	200	Bluish
							Hue

D5	4	<0.1	<0.1	8	7.5	300	Bluish
							Color

Kim '987 notes that the greenish color is eliminated and a neutrally colored material produced when the cobalt octoate is gradually added to blends of MXD6 and PET from a catalyst based on germanium and antimony or PET with virtually no residual cobalt and manganese (Col 4, lines 22-28).

- 11. I also conclude Kim '987 teaches away from the colored container. The claimed material of Kim '987 is the neutral color. One of ordinary skill knows that a neutral color is one without color. I conclude that Kim teaches the neutral color by limiting the amount of cobalt to 150ppm. (See col 4, lines 31 38 teaching that when the Cobalt is above 150ppm the blue begins to dominate the color of the blend and therefore 100-150ppm is the preferred range).
- 12. I have reviewed Kim 20020001684 (Kim '1684), the same inventor as in Kim '987. I conclude from these references that the color mentioned in the Office Action is not related to the domains but is caused by the catalyst residue. This is explicitly disclosed in Kim '1684, which claims priority from Kim '987. Kim '1684 states that, "It is known in the art that the color in the PET/MXD6 structures is due to the presence of catalyst residue in the polyester." [0050]. The color can be controlled by limiting the amount of catalyst. (Kim '1684, [0050]).
- 13. I also conclude that the Kim references teach to avoid green a color having both yellow and blue components. Both the Kim references teach to create a neutrally colored article, therefore as discussed in the First Examiner's Interview of application 10/769,167 (at which I was present), Kim does not teach towards colored containers. In fact, Kim '1684 teaches one

of ordinary skill to eliminate the visual colors [0038-0039].

- 14. The Office Action states that the addition of Cobalt Octoate eliminates the visual haze. However, this is not what I understand Kim to say nor what is demonstrated by the data. First, I want to eliminate the confusion as to the meaning of clarity in Kim '987. Kim '987 uses the word clarity to mean lack of color, not transparency or lack of haze, therefore whenever Kim uses the word clarity it should be taken to mean colorless, unless indicated otherwise. (See col 7, lines 46-50 and col 5, lines 37-38). Additionally, the first examples of Kim '987 are unoriented blends. Unoriented blends are not likely to have any visual haze caused by the domains. I know this based upon my direct observation of preforms containing polyester and MXD6. Alternatively, as taught in Kim '1684, biaxially oriented blends may have haze unless the degree of orientation does not exceed the limit at which the refractive characteristics of the blend materials change [0047].
- 15. I know that unoriented blends have spherical domains based upon my observation of SEM analysis and Kim '1684 [0030].
- 16. I also know that the monolayer containers A and B of Kim '987 are hazy. I know that because Kim '161 teaches that prior art containers are hazy (col 6, lines 31-32) and Kim '987 is one such prior art container. I know that stretched domains cause haze. As mentioned in my first Declaration, Kim '161 teaches that haze is caused by domains ranging from 2000 to 4000nm. This is not domains in the wavelength of light. I know this from my own observation and from Kim '161 which teaches that the orientation of PET and MXD6 during the manufacturing process (two stage reheat blow molding) results in the development of

haze caused by refractive index changes and the enlarged domains of MXD6 (col 8, lines 46 – 49).

17. I also know that the monolayer containers based upon Kim '987 are hazy based upon the data in Kim '987. I have recreated the data in Table II.

Table II

	PET Catalyst	Structure	Cobalt	Color	Transparent
A	Not Sb/Ge "not a/g-PET"	Monolayer	No	Green	
В	Not Sb/Ge "not a/g-PET"	Monolayer	100ppm	Dark Green	
С	Not Sb/Ge "not a/g-PET"	Multilayer 10% Core	No		Substantially
D	Not Sb/Ge "not a/g-PET"	Multilayer 10% Core	100ppm		Substantially
E	Not Sb/Ge "not a/g-PET"	Single Layer			

18. I know that the stretched monolayer container of Kim '987 is hazy and not transparent. I know this because Kim ''987 states that for the multilayer examples C and D, that "the improved optical properties (substantially transparent or translucent) is <u>due</u> to the fact that the blend layer [compositions A or B] is only 10% of the total structure" (col 8, lines 53 – 57). Thus, Kim '987 admits that A and B are not substantially transparent, but are in fact hazy. I also know that adding Cobalt made the container a darker green, yet the container was still considered "not transparent" or hazy. I also believe that the structures of Kim '987 A and B are hazy based upon the disclosures of Kim '1684 (table II) that teach that the injection-reheat blow process (stretch blow) has a normalized haze of 3.16% per mil equating to a haze value of 85% for the 27 mil bottle of Kim '987. This self reported poor optical property leads me to conclude that if there were domains between 400 and 700nm present in the container of

Example B, that the green and blue colors did not absorb enough light at the wavelengths corresponding to size of the domains so that X is less than 9.5 in the formula described in the specification.

- 19. I also note that the compositions used in the containers of Kim '987 are not the same formulation as Examples 1 D2 and 1-D3 which have the neutral color. The compositions used in the containers of Kim '987 are from Examples 2-A and 2-B which have the poor green color.
- 21. I have also reviewed Kim '987 to determine the distribution of the domains in the compositions, the number of domains at a given wavelength or the absorbance of light at wavelengths between 400 and 700nm. It cannot be done. I know that the domain size is determined by the injection or molding conditions and that the size can vary greatly. These variables are often inter-related and include, in part:
- a. surface tension between the two polymers,
- b. melt viscosity of the two polymers,
- c. the molecular weight of the two polymers (which influences the melt viscosity),
- d. the temperature at which the polymers are blended,
- e. the amount of work put into the polymer system which is a function of temperature, time, amperage and screw design,
- f. the amperage or draw of the blending device,
- g. the design of the screw,
- h. the residence time of the blend system in the blending device.

For instance, Al Ghatta et al EP0964031 [0029] reports domains as great as 1-2 micron in an unstretched article, while Al Ghatta WO03029349 (page 2, fourth paragraph) reports domain

distributions much less than 200 micron – all based upon extrusion conditions and shear. So, absent the extrusion conditions, I cannot, nor can anyone else, determine the size of the domains in the unstretched examples or the stretched examples of Kim '987.

- 22. I have also reviewed Kim '987 regarding whether adding cobalt to create a neutrally colored bottle would absorb enough light to prevent the scattering of light from domains, if present. First, I note that the colorless compositions are only D2 and D3 and I have no way of knowing whether there are domains between 400 and 700nm in that composition. I am very familiar with adding small amounts of cobalt based compounds, to tone out a color, typically yellow. This technique only adds enough cobalt so that relatively the same amount of light is reflected from the article. This is why Kim '987 limits the amount of cobalt to 150ppm so that the article does not become blue (col 4, line 32). Based upon my experience, the reading of Kim '987, and the specification, it cannot be determined that the composition without visual color (D2 and D3) has domains of MXD6 between 400 and 700nm, and if domains were present that enough light was absorbed corresponding to the size of the domains to achieve a value of X < 9.5. In fact, I would never expect it to.
- a. My experience with cobalt addition tells me that the amount of yellow being toned by 150ppm cobalt is very slight. Kim '987 supports this describing the color as "yellowish" (col 7, line 37). Blend D of Example 1 starts with blend B, which is stated as yellowish, meaning that it has only a slight amount blue absorbed, leaving slightly more yellow light to reflect than blue. Blend D2 contains 100ppm Cobalt and exhibits "improved clarity ... i.e. the yellow color appeared somewhat neutralized" (col 7, lines 47 48). Blend D3 at 150ppm Cobalt had a very neutral color. Blend D4 had 200ppm Cobalt and was considered

¹ I note that Kim '987 uses the word clarity to mean lack of color, not transparency or lack of haze, therefore whenever Kim uses the word clarity it should be taken to mean colorless, unless indicated otherwise.

to have a bluish hue, or light blue mask. Blend D5 had 300ppm and was considered a bluish color. All of this teaches one of ordinary skill is that Kim '987 is referring to slight color differences.

- b. My reading of Kim '987 teaches me that the amount of total light absorbed to create a neutral absorbance is slight. Kim '987 refers to the color being toned as yellowish and when twice the amount of cobalt is added, the color is a bluish color. I contrast this with the description of blend A being visually green (Example 1, and Example 2, bottle A).

 100ppm cobalt was added and the respective blend/bottle became a "pronounced green" (col 7, line 40) and dark green (*** Table 1). As discussed earlier I know bottle A and B to be hazy and non-transparent. I know that bottles A and B absorbed more light in the red region than did the yellowish and bluish hues of the neutrally colored bottle. I know this because green is made by adding a composition absorbing light in upper end of the red spectra (blue reflectance) and a composition or compound absorbing the lower end of the red spectra (yellow reflectance). Thus the green color and dark green absorb more than the yellowish/bluish combination. However, as discussed these bottles were hazy. Therefore, I can conclude that the composition with less light absorbed than a hazy bottle would not have a value of X less than 9.5.
- c. Comparison with specification: The specification teaches that a low amount of Tersar blue (0.05% was not enough to mask the visual haze and that 0.1% was, yielding an X value of 9.953 and 7.2 respectively (Table III). The spectra for the Tersar blues are given in Figure 12.
 - d. Conclusions: I can state that the addition of cobalt increases the amount of light

absorbed. However, because the article is colorless, e.g. not colored, or the opposite of a colored article as described in the claims, the only thing I can say is that it has enough light absorbed to offset a yellowish color. Because I know in relative terms that this not a dark color and is only hues, I would find it very surprising (again assuming there were domains

present in Ex-1 D2 and Ex-1 D3) if the X value is less than 9.5.

23. I was also present at the Examiner's Interview of patent application 10/769,167 where

two green bottles were shown. One green bottle was visually hazy, the other was not. These

bottles were of the exact same composition, same PET, same type and amount of MXD6 and

same amount of the same colorant. The difference between the bottles was that the

preforms from which the bottles were blown were manufactured on different injection

machines.

24. I declare that all statements made herein of my knowledge are true and that all statements

made on information and belief are believed to be true and, further, that these statements

were made with the knowledge that willful false statements and the like so made are

punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the U.S. Code

and that such willful false statements may jeopardize the validity of this application and any

patent issuing thereon.

Respectfully submitted,

Kev

Kevin L. Rollick, Ph.D.

Kevin J. Rollick

May 13, 2008

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